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FABRICATING REINFORCED PLASTICS BY FLUIDIZED BED TECHNIQUES

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BY

R. W. TOCK

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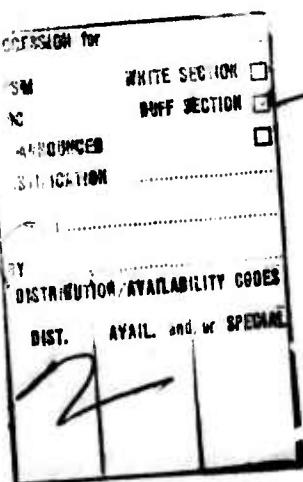
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FABRICATING REINFORCED PLASTICS BY FLUIDIZED BED TECHNIQUES

BY

R. W. TOCK

OCTOBER 1970

MONSANTO/WASHINGTON UNIVERSITY ASSOCIATION
HIGH PERFORMANCE COMPOSITES PROGRAM
SPONSORED BY ONR AND ARPA
CONTRACT NO. N00014-67-C-0218, ARPA ORDER 876
ROLF BUCHDAHL, PROGRAM MANAGER

MONSANTO RESEARCH CORPORATION
800 NORTH LINDBERGH BOULEVARD
ST. LOUIS, MISSOURI 63166

FOREWORD

The research reported herein was conducted by the staff of Monsanto/Washington University Association under the sponsorship of the Advanced Research Projects Agency, Department of Defense, through a contract with the Office of Naval Research, N00014-67-C-0218 (formerly N00014-66-C-0045), ARPA Order No. 876, ONR contract authority NR 356-484/4-13-66, entitled "Development of High Performance Composites."

The prime contractor is Monsanto Research Corporation. The Program Manager is Dr. Rolf Buchdahl (Phone-314-694-4721).
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FABRICATING REINFORCED PLASTICS BY FLUIDIZED BED TECHNIQUES

R. W. Tock

ABSTRACT

Fiber reinforced thermoplastics have been fabricated utilizing fluidized bed techniques. In this process the matrix is applied as a powder and fused to the surface of the fiber to provide a continuous coating. Composite test specimens are formed by compression molding. The work reported here was done primarily with a nylon 11 matrix and graphite fibers. Although scanning electron microscopy indicates difficulties in penetrating the multifilament graphite yarn with the polymer, the transverse mechanical properties of continuous fiber composites are quite high. The technique was also used with polycarbonate resin and reinforcements of glass fiber and stainless steel ribbon. The results indicate that a broad range of composite materials can be prepared using this technique.

Introduction

Continuous graphite fiber composites are generally formed with an epoxy resin because a thermosetting matrix offers several fabrication advantages compared to thermoplastic materials. With the epoxy, impregnation of the filler or roving is attained by a coating procedure, and this is normally followed by filament winding to form tapes, broad-goods or a finished geometry (1). If a thermoplastic resin is used instead of the thermoset, there are several approaches to fabricating the composites.

A coating-type operation for applying a thermoplastic requires either a polymer melt or a solvent solution. While the melt process permits the use of solvent intractable polymers, it also requires that they be thermally stable. In addition, the viscosity of a melt is usually so high that it is difficult to achieve good wetting and total fiber penetration. The difficulties encountered by high viscosity and viscoelastic polymer melts are reviewed in the recent paper by Harford and White (2). For example, their capillary rise measurements indicated that periods of up to two days were not uncommon for achieving complete flow equilibrium with melts at normal molding temperatures.

The solvent solution approach circumvents these problems, but creates others. First, a good casting solvent must be found, and then a process must be developed to remove it. In most instances it is safe to assume that the solvent is never completely removed. The question then arises as to the effect of the solvent on the polymer-graphite interface and ultimately on the performance of the composite. Second, the use of solvent invariably necessitates the need for greater safety precautions.

A third approach to the coating problem, the subject of this paper, is to fusion coat from a fluidized bed of the powdered polymer. This type of dry process coating has been used extensively to produce decorative and protective coatings on auto, machine, appliance and furniture parts (3,4). Fundamentally, it is similar to the polymer melt technique and hence has similar problems of wetting and penetration associated with high viscosity polymer melts. However, an important difference is that only the powder which comes in contact with the graphite is brought to the melt temperature so that thermal degradation problems are minimized. The polymer coated fibers are essentially prepreg materials which can be used for subsequent molding. A recent patent by Brooks (5) describes several fluidized polymer systems for coating continuous monofilament or sheet materials.

Here we describe the mechanical properties of composites formed using the fluidized bed technique. The success of the fluidized bed approach must ultimately be evaluated by a comparison of properties for composites made in this manner with those for composites fabricated by the alternate techniques for thermoplastics, especially the solvent solution approach. This will be the subject of a future paper. The experimental results described in this report are compared to data in the literature for graphite composites whenever possible. However, nearly all of the literature data is for different matrices and different fabrication techniques.

Experimental Considerations

The coating of sheet or wire materials using powdered polymers in fluidized beds is a standard commercial operation. Formulated powders for the process are readily available. The basic unit for the process consists of a fluidization chamber to contain the powder with equipment for the controlled passage of gas upward through the powder. The bed of powder expands, and in the dilated state it assumes fluid characteristics, i.e., it seeks its own level, flows into small openings, and offers little resistance to the insertion of solid objects.

Materials to be coated are usually heated to a temperature above the fusion point of the polymer, dipped into the bed, withdrawn, and cooled to room temperature. In some instances a solvent precoat or an electrostatic process is used to promote adhesion of the powder to the substrate when it is dipped into

the bed. In this case, a final heating cycle to fuse the adhering powder is also required.

The degree of surface coverage and polymer buildup produced in each coating operation is a function of several variables. These variables have been defined and their interrelationships published in the literature (4,5,6). Within the limitations imposed by time and materials when the object is preheated prior to dipping, a high initial temperature, large heat capacity, good heat transfer coefficient, or long immersion time will all increase the amount of polymer fused or built up. Less obvious are the effects of gas velocity through the bed and the particle size of the powder. Here again, within limits, the faster air velocities and smaller particles will both produce thinner coatings. Practical bounds have been established at 1 to 3 feet per minute for gas velocities.

With the small diameter fibers encountered in this experiment, it was considered impractical to heat the fiber prior to bed insertion. Instead, several attempts were made at heating the graphite yarn as it passed through the bed. For a batch system, heating the graphite by electrical resistance was acceptable. However, in the continuous coating operation we were unable to maintain a good electrical contact downstream because of the insulating coating of polymer on the graphite.

The approach finally selected was a pulling of the yarn through the bed and allowing it to pick up a powder coating

through simple electrostatic attraction. The coated yarn was drawn immediately through a tubular furnace where the powder fused to a continuous film. With the graphite yarn this approach was workable, but the powder coating was, at best, a fragile one. Moreover, the rate at which the yarn was coated was now dependent on how well the fiber picked up the powder. Under these circumstances powders with smaller particles were more effective, while the gas flow rate had little or no apparent effect on the degree of coating.

In the initial phases of this experiment, continuous graphite yarn (Hitco, HMG-50) was selected for coating. This is a two-ply bare yarn with 720 filaments per ply twisted to four turns per inch and having an individual filament diameter of 2.7×10^{-4} inch. The coating apparatus is shown schematically in Figure 1. On a typical run the yarn was stripped from the feed reel, 1, and passed through a 0.1% solution of a silane coupling agent (Dow Corning, Z-6050), 2, in methanol. Although the silane is reactive with nylon (7), it was not used primarily as an adhesion promoter to graphite, but more as a cleaning solution for the graphite and for improved wetting at the interface.

The graphite yarn was then taken through the fluidized bed, 3. Here a nitrogen gas stream, 4, directed upward at 2 to 2.5 feet per minute through the porous glass bottom

of the chamber produced a dense phase fluidization of the nylon 11 powder, 5. This powder is a specialty product (Aquitaine Chemical Co.) specifically for fusion coatings from fluidized beds. As the graphite yarn passed through the bed, the powder was attracted to the graphite by electrostatic forces.

From the bed the powder-covered yarn was drawn through a tubular furnace, 6. Here the powder fused to the graphite, forming a continuous film. For this particular combination of polymer and fiber, a furnace wall temperature of 250°C and a residence time of 15 seconds were required. Longer times and higher temperatures appeared to cause polymer degradation while shorter times and lower temperatures produced inadequate fusion. Once the yarn was coated it was wound on a take-up drum, 7, and stored for later fabrication.

To evaluate the mechanical properties of the coated yarn, continuous and short (chopped) fiber composites were formed by compression molding. Basic plate geometries with a thickness dimension of 1/16 inch were fabricated. Press platen temperatures of 575°F and a pressure of 100 psia were applied to close the mold. When the mold was closed to the predetermined sample thickness, the cooling cycle was started and the pressure increased to 8000 psia. Uniaxial alignment was assumed when all the coated yarns were continuous and assembled with one principal orientation prior to molding. The random samples were composed of

1/2" long chopped fiber bundles randomly dispersed in the mold cavity before molding. These samples showed no preferred orientation. Coupons were cut from the molded sheets for tensile testing in the Instron and tabs were positioned at the ends of the samples to help dissipate clamping stresses. Injection molded samples were also prepared using coated fiber chopped to a length of 1/4". The barrel and mold temperatures were 530°F and 180°F, respectively.

Tensile properties were determined at a crosshead speed of 0.05 in./min. for all samples, and strain was monitored with a one inch gage length extensometer. After testing to failure, selected samples were subjected to the following analyses: measurement of volume loading (8) and fiber orientation distribution (9), and the evaluation of the fracture surfaces with the electron scanning microscope (9,10).

Experimental Results

Tensile properties are listed in Table I for the graphite-nylon 11 composites fabricated with the fusion coated yarn. The longitudinal stiffness values are within five percent of the rule of mixtures. The transverse modulus is comparable to literature values, and the modulus for the chopped random fiber material is close to the prediction for isotropic stiffness by Tsai and Pagano (11).

Strength values of the aligned compression molded samples are the principal criteria for evaluating the fabrication technique. The extent of wetting and adhesion achieved between polymer and fiber are determined by fracture surface examination and an analysis of transverse and shear strength values. The transverse strength of continuous fiber samples approaches the yield strength of the resin. Further, this value is close to the 90° strength requirement (8000 ksi) for Air Force graphite composites (12). The shear strength data were hard to classify since it was never apparent whether failure was due to shear of the composite or yielding of the low modulus nylon 11. An average value for the shear experiments was 5.3×10^3 psi.

The strength values of the continuous longitudinal samples are very similar to data for continuous graphite-epoxy composites (13). Electron scanning micrographs of the fracture surfaces in Figures 2 and 3 indicate the existence of good adhesion (9) in localized regions, and poor adhesion in other areas. Microscopic evaluation of the coated yarn prior to compression molding revealed many instances of poor polymer penetration. This is shown in Figure 4 with the yarn appearing as a sheathed cable. Examination of composite fracture surfaces indicates that during the molding operation nearly complete penetration must occur.

The tensile strength data for the random fiber samples are also listed in Table I. Currently, the literature does not list strength data for random graphite-epoxy composites. The results are significantly higher than values reported for random graphite-polycarbonate composites (14).

Property levels for the injection molded tensile specimens were comparable to those obtained with a commercial graphite-nylon 6/6 molding compound (Hercules). However, the fibers in the commercial molding material are completely wetted prior to the injection molding operation, whereas the chopped fusion coated graphite definitely is not totally wetted before molding.

To demonstrate the flexibility of a fluidized bed technique, S-glass strand, i.e., bundled fibers with 0° twist, and stainless steel ribbons (Olin Brass Co., Type 302) were coated with nylon 11 in the manner previously described for the graphite yarn. In addition, composites were made using graphite (Union Carbide, Thornel 50) coated with polycarbonate (General Electric, Lexan). The mechanical properties for composites formed with these materials are listed in Table II.

The ribbon was used as an example of coating a reinforcing material without penetration problems. In this case the coating was very similar to the process described by Brooks. The strength and modulus results are very close to the values obtained with stainless ribbons and a polyethylene ionomer matrix (15).

The coating of the S-glass differed from the coating behavior of the graphite since there was zero twist to the glass. This made penetration by the polymer easier since the electrostatic charge essentially opened the fiber bundle in the bed and permitted considerably thicker coatings of polymer on the glass. After application of heat in the tubular furnace, the polymer did not appear as a sheathed cable as with graphite, but there was much greater penetration and wetting. The mechanical property levels are lower than literature values for S-glass-epoxy composites (16,17).

Although only preliminary work was carried out on the polycarbonate-graphite system, it was evident that coating with this polymer was more difficult than with nylon 11. The higher viscosity of the proportionate melt caused a greater problem of penetration of the resin into the twisted graphite. However, the longitudinal tensile properties of aligned compression molded composites were nearly equal to the values for Hitco graphite and nylon 11.

DISCUSSION

The use of a fluidized bed to coat reinforcing fibers and form prepreg material should ideally be carried out with the fiber heated to the melt temperature of the polymer. In this way polymer penetration takes place within the bed, but here penetration was accomplished in the molding operation. As a result,

complete penetration is quite unlikely. However, the transverse strength of composites formed by graphite and nylon 11 approach the yield strength of the resin which indicates that nearly complete wetting must have been attained.

Two factors to be noted concerning the coating process are the air velocity used to expand the bed and the particle size of the powdered polymer. The air velocity did not seem to affect the thickness of the coating, provided the flow rate was sufficient to maintain the bed in the dense phase. However, very fast rates did cause channeling and elutriation to occur.

Particle size seemed to have an effect on both the quality and thickness of the coating. In all cases the finer powders gave a better product. They were more susceptible to agglomeration and channeling, but this could be overcome by vibrating the bed. Photographs and size distributions of the nylon 11 and polycarbonate powders used are shown in Figures 5 and 6. Their morphologies are quite different, which was attributed to their method of manufacture. The nylon shows the sharp edges produced by grinding at cryogenic temperatures, while the embryonic configuration of polycarbonate is typical of formation in solutions. The size distribution of the smaller nylon powder is also much narrower, due to better screening. However, only the size of the powder, not the shape, affects the coating process.

In the analysis of the mechanical properties, it could not be determined if the measured longitudinal continuous fiber strength values were lower than the actual values because of experimental conditions. There was a problem of adhesion of tabs to the composite test specimens, and about half of the samples failed at the tabs (jaw break). However, as indicated above, the measured values are close to the values in the literature for graphite-epoxy.

The chopped random graphite composites were formed by randomizing chopped coated yarns and compression molding. This fabrication procedure minimizes breakage of the fiber. This is indicated by the high values of tensile strength and some limited analyses of fiber length distribution on the fractured specimens. On the other hand, fiber breakage was severe for the injection molded samples. Also, scanning electron micrographs of the injection molded fracture surfaces indicate that polymer wetting was not as complete as with the compression molded samples. The data for the graphite-nylon 11 injection molded composite, when compared to injection molded glass filled thermoplastics(18), indicate about equal strength, but higher modulus for the graphite even with the extensive fiber damage.

SUMMARY

The fluidized bed coating technique is a method of making composite prepgs using thermoplastic resins. It is especially applicable when the polymer cannot be solvent coated on the fiber. The process has an advantage over other melt coating operations since the problem of polymer degradation is minimized. Also, the process could be used with thermosets, although such work is not reported here.

The mechanical properties for composites formed from the prepreg are significantly high in two cases: the transverse strength data of the continuous aligned graphite-nylon 11 system; the strength and modulus of the chopped randomly oriented graphite-nylon 11 material.

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TABLE 1

TENSILE PROPERTIES OF HMG-50—NYLON 11 AT 25°C

<u>Constituents:</u>	HMG-50	Nylon 11
strength (10^3 psi)	300	7.85
modulus (10^6 psi)	50	0.185
elongation (%)	0.6	300

<u>Composites:</u>	<u>Experimental Data*</u>	<u>Comparative Values</u>
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<u>continuous aligned/compression molded</u>	<u>continuous HMG-50—epoxy</u>
--	--------------------------------

	$V_f = 0.50$	V_f		reference
		94	110	
longitudinal	strength (10^3 psi)	94	110	(13)
	modulus (10^6 psi)	23	28	
transverse	strength (10^3 psi)	6.5	2	(13)
	modulus (10^6 psi)	1.6		

<u>discontinuous random/compression molded</u>	<u>random HMG-50—polycarbonate</u>
--	------------------------------------

	$V_f = 0.50$	$V_f = 0.20$	reference
strength (10^3 psi)	22	16.3	(14)
modulus (10^6 psi)	6.5	3.8	(14)

<u>discontinuous/injection molded</u>	<u>discontinuous/injection molded</u>
---------------------------------------	---------------------------------------

<u>Hercules molding compound</u>	<u>(nylon 6,6 matrix)</u>
----------------------------------	---------------------------

	$V_f = 0.30$	$V_f = 0.20$
strength (10^3 psi)	15	17
modulus (10^6 psi)	2.4	1.4

*Average values, except for longitudinal strength (jaw breaks not included in averaging).

TABLE 2
TENSILE PROPERTIES

Constituents:

	strength (10^3 psi)	modulus (10^6 psi)
S glass	500	12
Stainless steel	170	29
Thornel 40	250	40
Nylon 11	7.85	0.185
Polycarbonate	9.5	0.35

Composites:

Experimental Data*		Comparative Values	
S glass—nylon 11 continuous aligned/compression molded	$v_f = 0.45$	S glass—epoxy	
longitudinal strength (10^3 psi)	108	$v_f = 0.716$	reference
modulus (10^6 psi)	5.5	275	(16)
transverse strength (10^3 psi)	5.0	9.6	(16)
modulus (10^6 psi)	0.5	8.0	(17)
discontinuous random/compression molded	$v_f = 0.25$		
strength (10^3 psi)	28		
modulus (10^6 psi)	1.6		
stainless steel ribbons—nylon 11		stainless steel ribbons—ionomer	
transverse strength (10^3 psi)	$v_f = 0.50$ 43	$v_f = 0.52$	reference
modulus (10^6 psi)	11.6	40	(15)
random strength (10^3 psi)	$v_f = 0.70$ 62.4	9.5	(15)
modulus (10^6 psi)	14.2		
Thornel 40/polycarbonate continuous aligned/compression molded		unidirectional lamina	
strength (10^3 psi)	$v_f = 0.50$ 75.6	$v_f = 0.64$	reference
modulus (10^6 psi)	19.6	130	(13) Thornel 50-epoxy
		19.2	(19) Thornel 40-epoxy

*Average values, except for longitudinal strength (jaw breaks not included in averaging).

FIGURE 1

SCHEMATIC OF FUSION COATING APPARATUS

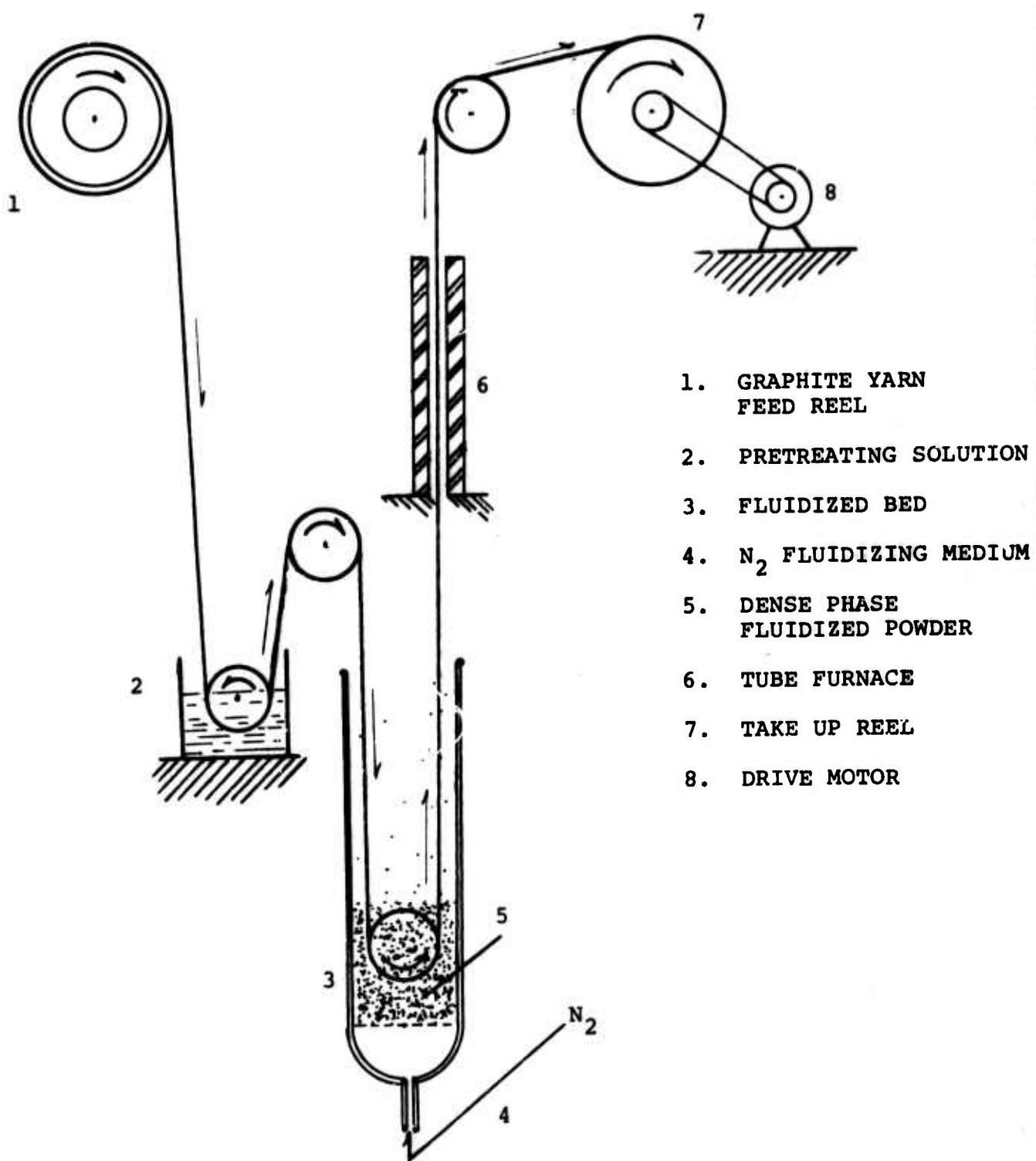




Figure 2 Good Adhesion

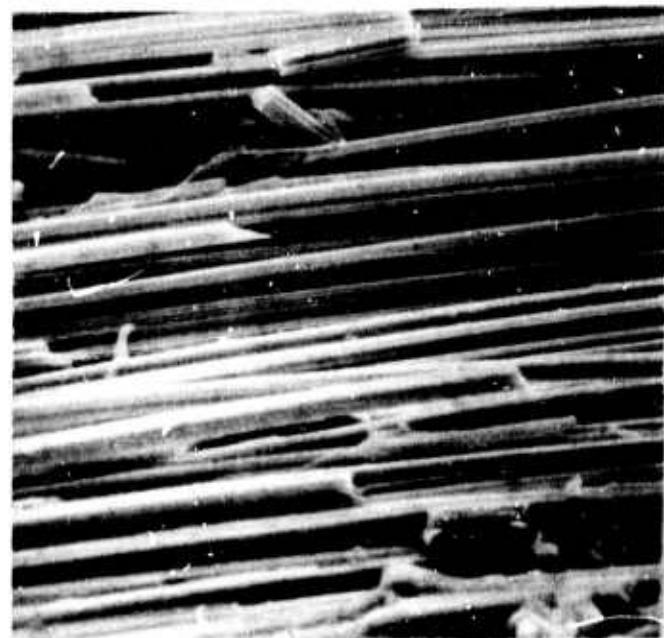


Figure 3 Poor Adhesion



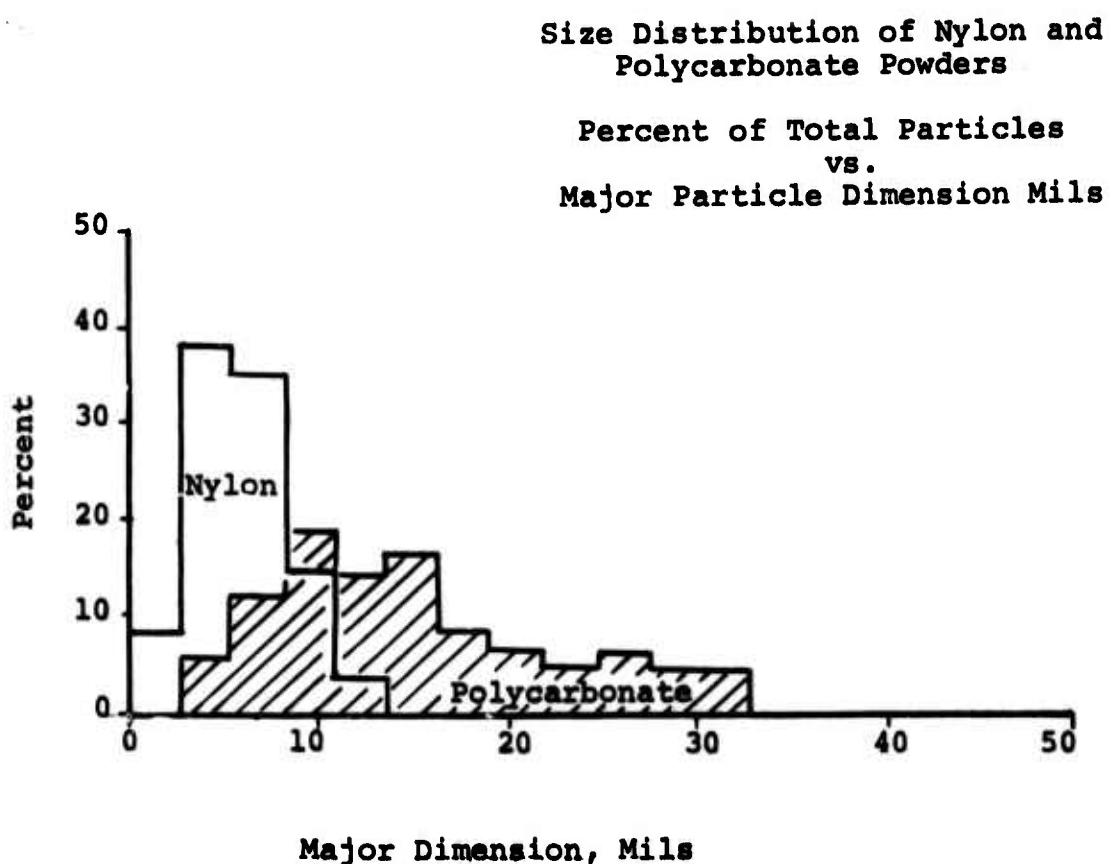
Poorly Coated Graphite



Adequately Coated S-Glass

Figure 4

NOT REPRODUCIBLE



NOT REPRODUCIBLE

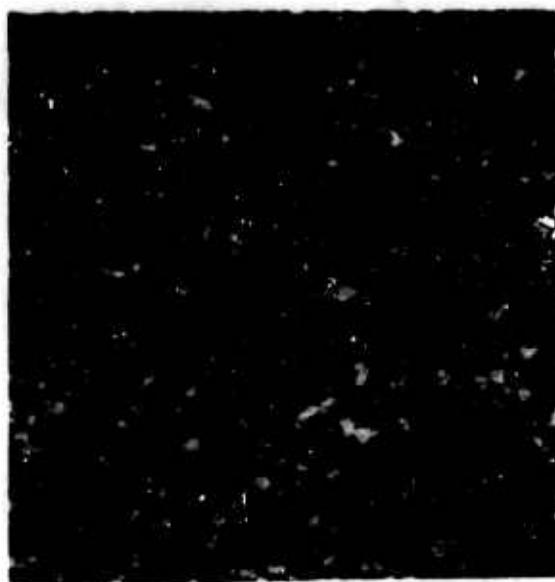


Figure 5 Nylon 11 Powder
X13

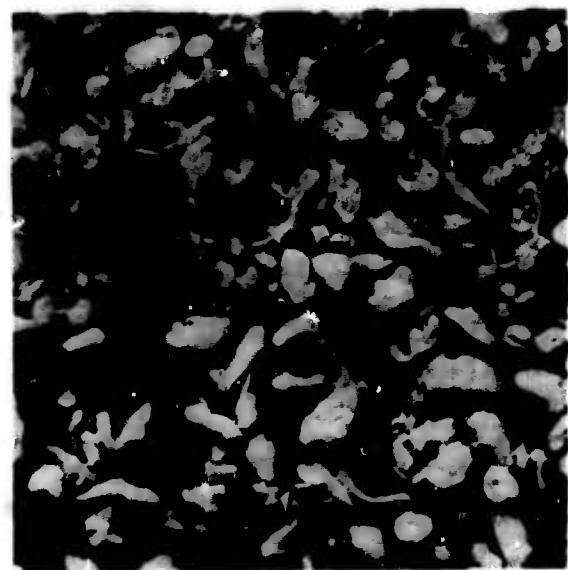


Figure 6 Lexan Polycarbonate Powder X13

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13. ABSTRACT
 Fiber reinforced thermoplastics have been fabricated utilizing fluidized bed techniques. In this process the matrix is applied as a powder and fused to the surface of the fiber to provide a continuous coating. Composite test specimens are formed by compression molding. The work reported here was done primarily with a nylon 11 matrix and graphite fibers. Although scanning electron microscopy indicates difficulties in penetrating the multifilament graphite yarn with the polymer, the transverse mechanical properties of continuous fiber composites are quite high. The technique was also used with polycarbonate resin and reinforcements of glass fiber and stainless steel ribbon. The results indicate that a broad range of composite materials can be prepared using this technique. ()

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Ribbons						
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Compression molding						
Mechanical properties						

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